

14.GS:
CIR 289
C. 2

Geol Survey

STATE OF ILLINOIS
WILLIAM G. STRATTON, *Governor*
DEPARTMENT OF REGISTRATION AND EDUCATION
VERA M. BINKS, *Director*



Effect of High-Carbon Components and Other Additives on the Character of Cokes

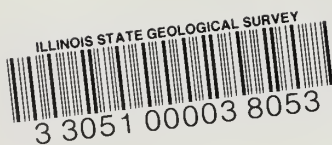
Laboratory-Scale Study

John A. Harrison

DIVISION OF THE
ILLINOIS STATE GEOLOGICAL SURVEY
JOHN C. FRYE, *Chief* URBANA
CIRCULAR 289 1960

ILLINOIS GEOLOGICAL
SURVEY LIBRARY

MAR 31 1960



EFFECT OF HIGH-CARBON COMPONENTS AND OTHER ADDITIVES ON THE CHARACTER OF COKES

LABORATORY-SCALE STUDY

John A. Harrison

ABSTRACT

Mixtures of a standard coal sample (70 percent Illinois No. 6 Coal - 30 percent Illinois No. 5 Coal) with fusain, anthracite, coke dust, petroleum coke, black shale, special prepared sizes of the standard blend, and a benzene extract of coal tar pitch ("resin"), under certain conditions, produced cokes of higher quality than coke made from the standard sample alone, as determined by laboratory-scale coke tests.

Improvement varied depending upon an interrelation between nature, size, and amount of additive used. Fusain or anthracite, plus "resin," added to the standard sample produced the best cokes of the test series.

INTRODUCTION

Most metallurgical coke is produced from blends of two or more coals to give it the desired physical properties. The marked decline in reserves of the best coking coals, plus certain economic factors, have encouraged blending some coals which formerly were regarded as marginal coking coals. An understanding of the physical and chemical properties of such coals is necessary in order to produce an optimum metallurgical coke. A number of laboratories, some with pilot-scale facilities, are conducting coking and other tests on these marginal metallurgical coking coals. Several publications related to this general subject and published by members of the Illinois State Geological Survey are given in the references of this paper.

Laboratory investigations, at the Illinois State Geological Survey, of the Illinois No. 6 and No. 5 Coals have indicated that the petrographic composition of the coal charge may be an important influence on character of coke produced (Marshall et al., 1958). Pilot-scale tests in which the petrographic composition of the coal charge was altered by increasing the amount of the high-carbon component fusain, have been made with Illinois coals. The fusain was hand-picked from No. 6 Coal.

Although direct correlation between the laboratory and pilot-scale tests could not be made, the trends established in tumbler and shatter indices of the cokes made in the laboratory were generally the same as those from the pilot-scale tests. In both laboratory and pilot-scale tests certain physical properties of the resulting coke were improved by the addition of an optimum amount of fusain.

The present study concerns the influence upon the physical properties of coke produced by adding high carbon and other components to a standard sample of Illinois No. 6 and No. 5 Coals. Laboratory-scale coke tests established an optimum blend of these two coals (70 percent No. 6 Coal - 30 percent No. 5 Coal) which was used as the standard sample throughout the investigation. The high carbon and other components, hereafter referred to as "additives" in this paper, varied in amounts, size, and chemical composition. Almost all the additives except the black shale contained a higher carbon content than the coal. Trends established in these laboratory studies, in which the petrographic composition of the charge to be coked (standard sample plus additive) was closely controlled, provide a basis for pilot-scale tests.

Acknowledgments

Various coal companies furnished the coals and other components that were blended with the coal in this laboratory coking investigation. The column sample from the No. 5 Coal was obtained from the Sahara Coal Company Mine No. 16, Saline County, Illinois. Coal samples and fusain were taken from the No. 6 Coal of the Freeman Coal Mining Corporation Orient No. 3 mine, Jefferson County, Illinois. Anthracite was furnished by the Glen Alden Corporation, Hudson Coal Company, and Jeddo-Highland Coal Company. The Great Lakes Carbon Company supplied the petroleum coke for the investigation. The coal tar pitch, from which the "resin" was derived was supplied by Inland Steel Company. The black shale was obtained from a United Electric Coal Company strip mine in Fulton County.

G. R. Yohe, Head of Coal Chemistry Section of the Geochemical Group, Illinois State Geological Survey, prepared the "resin" extract from the coal tar pitch.

PROCEDURES, PREPARATION, AND ANALYSIS

Coal Samples

For comparison with previous laboratory coking investigations (Marshall et al., 1958) it was desirable to obtain coal samples representative of the coals used in the earlier study. Channel samples, six inches wide and four inches deep, were cut from the fresh face of No. 6 Coal in Jefferson County and from No. 5 Coal in Saline County. Bands of mineral matter $\frac{1}{4}$ -inch or larger were removed and the samples were sealed in air-tight cans in the mine to minimize oxidation.

Previous studies demonstrated a method of crushing which yielded optimum physical properties in the coke produced from the No. 6 and No. 5 Coals on a laboratory scale. This crushing procedure, adopted for the present investigation, consisted of the following steps: the sample was passed over a screen with a mesh opening of 3 mm ($1/8$ inch); the undersize material made up the first portion of the final sample. The oversize material was passed once through a jaw crusher (jaw separation approximately $3/4$ inch). The crushed material was passed over the screen again and the undersize added to the final sample. The oversize material was slowly passed through a small set of roll crushers set at a roll separation of 3 mm, and again screened at 3 mm. The undersize was added to the final sample and the oversize repassed through the rolls set at 3 mm. The cycle was repeated three times after which only about 0.3 of one percent of the material exceeded the 3 mm size. So far as practicable, all coal dust was collected and added to the final sample.

Table 1. - Size Analysis of Coal and Additives used in Laboratory Coking Tests

Sample	Tyler Standard Screen Scale Sieves $\sqrt{2}$							Pan
	6	10	20	35	65	150	270	
No. 6 Coal	0.2	40.9	29.1	13.8	7.1	4.8	1.1	3.0
No. 5 Coal	0.3	39.8	29.6	14.8	8.9	2.8	1.3	2.5
Anthracite -10 mesh		0.0	11.7	36.3	28.6	14.5	3.7	5.2
Anthracite -20 mesh			0.4	41.7	33.0	16.2	3.8	4.9
Coke dust -20 mesh			0.0	2.1	10.7	37.2	25.7	24.3
Coke breeze -10 mesh		0.3	31.2	25.0	17.9	16.1	4.7	4.8
Coke breeze -20 mesh			0.4	33.2	28.6	23.8	6.5	7.5
Petroleum coke -10 mesh		0.2	27.8	21.2	16.0	15.6	9.3	9.9
Petroleum coke -20 mesh			1.3	24.0	23.9	22.1	13.4	15.3
Black shale -10 mesh		0.2	31.5	28.2	17.7	12.8	3.5	6.1
Black shale -20 mesh			3.2	43.8	23.7	16.3	4.6	8.4
"Fine coal" -10 mesh		0.2	42.1	26.1	13.8	9.3	2.7	5.8
"Fine coal" -20 mesh			1.1	40.1	25.4	17.9	5.0	10.5
-48 mesh coal dust				32.7	18.4	24.1	9.2	15.6
			Tyler $\sqrt{2}$ Sieves					
			150	170	200	250	270	Pan
Fusain -150 mesh			0.7	0.4	2.2	4.2	2.2	90.3
Anthracite -150 mesh			0.6	1.4	2.7	0.6	19.9	74.8
Coke dust -150 mesh			1.1	0.8	1.7	1.4	3.9	91.1
Black shale -150 mesh			3.4	0.7	4.2	7.2	25.2	59.3
"Fine coal" -150 mesh			1.6	0.3	2.4	1.6	33.3	60.8

Each coal sample was thoroughly mixed, quartered to give approximately 1200-gram samples, and stored in air-tight cans. The samples of No. 6 and No. 5 Coals had essentially the same size composition (table 1 and fig. 1). This was accepted as the basic size composition and was used consistently throughout the test program.

To prepare the broken coal for petrographic analysis, it was mixed with an equal amount of paraplex (Paraplex "P" series resins) in a paper container and cured in an oven at 90°C. for 8 hours. After curing, a flat surface was ground using No. 3 dry emery paper. A finer 3/0 dry emery paper used in the second grinding produced a flat, partially polished surface. The third and fourth steps were carried out on polishing wheels covered with "metcloth" (nap-free cloth) and using an aqueous suspension of No. 1 and No. 3 alumina respectively, with a minimum amount of water. In the final stage the sample was polished on a high quality billiard cloth which was washed continuously with a fine stream of distilled water. This method produced a highly polished, scratch-free surface.

For this investigation macerals were designated as vitrinite, exinite, semi-fusinite, and inertinite. Vitrinite is a group term which includes all vitrain with the lower limit imposed by the resolving power of the microscope, humic degradation matter, resin rodlets, and resins classified as red resins in transmitted light studies. All spore coats, cuticles, and yellow resins were considered as exinite. Semi-fusinite consisted of material intermediate between vitrinite and fusinite.

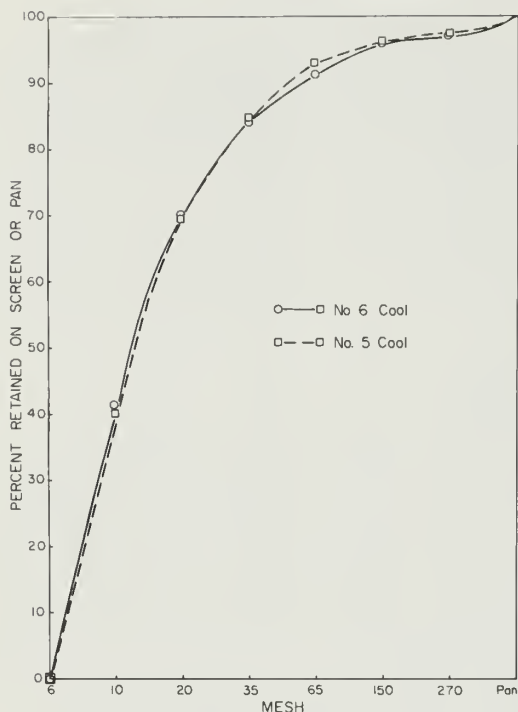


Fig. 1. Size analysis of No. 6 and No. 5 Coals.

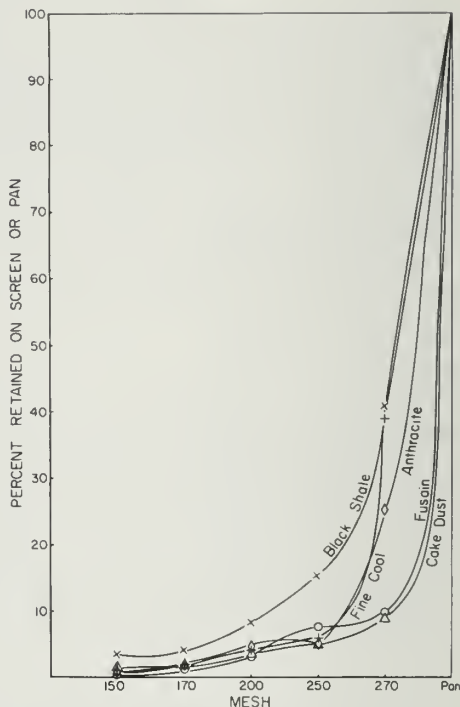


Fig. 2. Size analysis of minus 150-mesh additives used in coal blends for the production of laboratory coke.

Under the term inertinite were grouped the macerals known as fusinite, micrinite, and a material that resembled sclerotinite. Visible mineral matter was also determined.

Petrographic analyses of broken coal samples were determined by the point-count method (Chayes, 1949). The analyses were made on polished surfaces using a microscope equipped with a vertical illuminator and oil immersion objective. Magnification was 320 diameters.

Shape analyses were determined using the same microscope but with an optical system having a dry objective and magnification of 128 diameters. Criteria established for the shape analyses placed individual coal particles in one of five categories - equi-dimensional, elongate, rodlike, triangular, and angular. Equi-dimensional particles varied in shape from round to square, although all particles had two axes essentially the same length in the plane of the polished surface. Elongate particles were those in which one axis on the flat surface was longer than the second but less than twice as long. In the rodlike classification one axis of the particles was from more than twice as long to many times as long as the second axis. Triangular particles were those that had three sides. Those particles that had many sides and no definite geometric figure in which the position of two axes could be determined were designated as angular.

Petrographic analyses of the No. 6 and No. 5 Coals, used in this investigation, were essentially the same. The No. 5 Coal had a slightly higher amount of semi-fusinite and slightly lower amount of exinite than the No. 6 Coal, but otherwise little difference was found (table 2). Petrographic analyses of the seven blends used in establishing the standard sample were similar (table 2 and fig. 6).

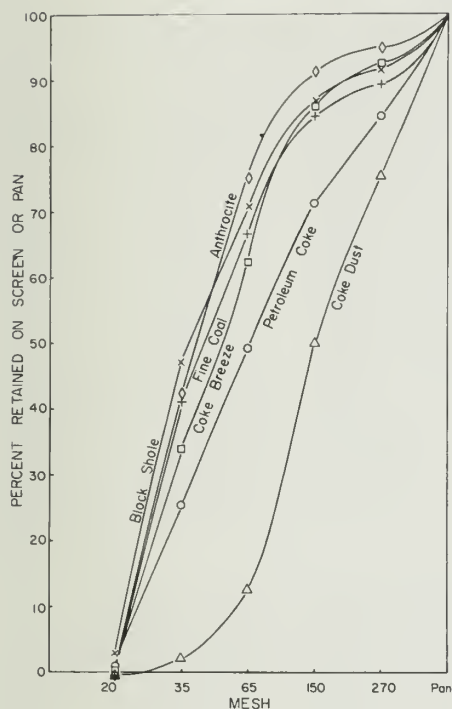


Fig. 3. Size analysis of minus 20-mesh additives used in coal blends for the production of laboratory coke.

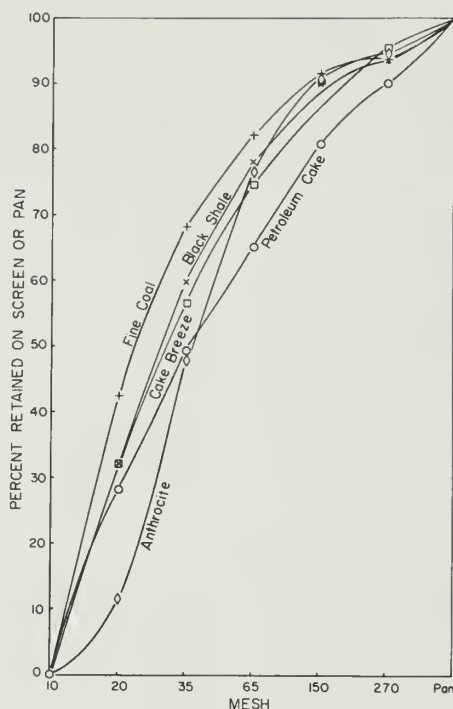


Fig. 4. Size analysis of minus 10-mesh additives used in coal blends for the production of laboratory coke.

The percentage of inertinite and visible mineral matter varied only 0.1 throughout the different blends as shown by the analyses (table 2 and fig. 6). Vitrinite and semi-fusinite showed an increase and exinite showed a decrease throughout the blends from 100 percent No. 6 Coal to 30 percent No. 6 Coal.

Chemical analysis of each coal demonstrated that the two coals are similar (table 3). The similarity of the two coals also was observed in a study of the shape analyses (table 4).

Additives

Additives used in this investigation were fusain, anthracite, coke dust, coke breeze, petroleum coke, minus 48-mesh Illinois coal from a commercial preparation plant, blend of No. 6 and No. 5 Coals reduced to a size smaller than the basic size composition and designated as "fine coal," black shale from western Illinois, and a benzene-soluble, white-gasoline-insoluble extract of coal tar pitch called "resin" or "asphaltene." Additives were prepared in three size ranges of definite size composition whenever possible: minus 150-mesh, minus 20-mesh, and minus 10-mesh. Breakage characteristics and degree of previous preparation of some additives prevented as close control as was possible for the coal. Table 1 and figures 1 through 4 give the size analyses of all components of coals and additives used in this investigation.

Table 2. - Petrographic Analysis of Coals, Additives, and Certain Blends Used in Laboratory Coking Tests

Sample	Vitrinite	Exinite	Semi fusinite	Inertinite	Mineral matter (visible)	Vitrinite*
No. 6 Coal	82.3	8.3	0.5	6.8	2.1	
No. 5 Coal	83.8	6.3	1.1	6.7	2.1	
Fusain -150 mesh	2.2	0.4	0.0	96.1	1.3	
Fusain 20 x 150 mesh	6.8	0.2	0.2	82.0	10.8	
"Fine coal" -10 mesh	82.0	6.3	1.0	7.3	3.5	
"Fine coal" -20 mesh	80.8	6.1	0.9	8.4	3.8	
"Fine coal" -150 mesh	87.5	4.9	0.0	6.3	1.3	
Anthracite -10 mesh				7.3	0.9	91.8
-48 mesh coal dust	79.6	3.4	← 13.2 →		3.8	
Blends of						
No. 6 Coal	No. 5 Coal					
%	%					
100	0	82.3	8.3	0.5	6.8	2.1
90	10	82.4	8.1	0.6	6.8	2.1
80	20	82.6	7.9	0.6	6.8	2.1
70	30	82.7	7.7	0.7	6.8	2.1
60	40	83.0	7.5	0.7	6.8	2.0
50	50	83.0	7.3	0.8	6.8	2.1
40	60	83.2	7.1	0.9	6.7	2.1
30	70	83.3	6.9	1.0	6.7	2.1

* Vitrinite material from a higher rank coal. Fixed carbon is 92-98 percent.

Table 3. - Chemical Analysis of Coals and Additives Used in Laboratory Coking Tests

Sample	As Received Proximate					Moisture- and Ash-Free Proximate			
	Moist.	V.M.	F.C.	Ash	Total sulfur	V.M.	F.C.	Btu/lb.	Total sulfur
No. 6 Coal	9.6	32.7	50.4	7.3	0.91	39.4	60.6	14,299	1.09
No. 5 Coal	7.9	33.9	50.4	7.8	1.30	40.2	59.8	14,615	1.54
"Fine coal" -20 mesh	8.6	33.9	49.6	7.9	1.05	40.7	59.3	14,350	1.25
"Fine coal" -150 mesh	5.8	33.0	53.3	7.9	1.13	38.2	61.8	14,317	1.30
Fusain -150 mesh	0.7	9.8	81.4	8.1	2.68	10.7	89.3	15,076	2.94
Fusain 20 x 150 mesh	0.7	11.7	68.7	18.9	8.64	14.6	85.4	14,535	10.75
Anthracite -20 mesh	1.6	8.5	82.2	7.7	0.71	9.4	90.6	15,046	0.78
Anthracite -150 mesh	1.9	6.2	83.9	8.0	0.73	6.9	93.1	15,083	0.81
Petroleum coke -10 mesh	0.3	13.5	86.1	0.1	0.83	13.6	86.4	15,790	0.83
Coke dust -20 mesh	1.5	1.8	87.6	9.1	0.77	2.0	98.0	14,211	0.86
Coke breeze -20 mesh	1.9	4.6	78.8	14.9	0.98	5.5	94.5	14,124	1.18
Black shale	3.5	23.2	13.1	60.2	1.52	63.9	36.1	12,736	4.19
Coal dust -48 mesh	4.7	32.2	51.0	12.1	0.84	38.7	61.3	14,282	1.01
"Resin"	2.5	50.5	47.0	0.04	0.48	51.8	48.2	15,929	0.49

Table 4. - Shape Analysis of Coals and Additives used in Laboratory Coking Tests

Sample	Equal dimensional	Elon-gate	Rod-like	Tri-angular	Angular	Elongate + Rod-like
No. 6 Coal	2.9	42.0	44.7	7.5	2.9	86.7
No. 5 Coal	2.5	40.9	48.8	5.1	2.7	89.7
Fusain -150 mesh	5.3	9.2	79.6	3.4	2.5	88.8
Anthracite -10. mesh	0.8	52.7	36.1	7.4	3.0	88.8
Anthracite -150 mesh	8.5	60.9	21.9	6.8	1.9	82.8
Coke dust -20 mesh	2.6	54.6	32.3	6.7	3.8	86.9
Coke dust -150 mesh	8.5	46.2	36.1	6.7	2.5	82.3
Coke breeze -20 mesh	1.9	48.5	29.6	4.2	15.8	78.1
Petroleum coke -10 mesh	4.5	71.1	14.8	7.6	2.0	86.5
Petroleum coke -20 mesh	2.6	68.7	21.2	5.7	1.8	89.9
"Fine coal" -10 mesh	16.6	38.2	32.8	9.0	3.4	71.0
"Fine coal" -20 mesh	0.8	39.9	51.5	5.9	1.9	91.4

Fusain

Lenses of almost pure fusain four to five inches thick were handpicked from the No. 6 Coal bed, dried, and screened without crushing. Hard mineralized fusain was retained on the 20-mesh screen. Screen analysis of the minus 150-mesh fusain showed that the natural breakage of this material during the screening process formed a product in which 90.3 percent was smaller than minus 270-mesh (table 1 and fig. 2).

Petrographic analyses proved that the inertinite content is higher and the visible mineral matter lower in the minus 150-mesh fusain than in the basic size composition of both coals (table 2). A comparison between the 20 x 150-mesh fusain and the minus 150-mesh fusain showed that the visible mineral matter in the latter decreased from 10.8 to 1.3 percent, vitrinite decreased from 6.8 to 2.2 percent, but inertinite increased from 82.0 to 96.1 percent. Differences are also demonstrated in the chemical analyses when ash, total sulfur, volatile matter, and fixed carbon are inspected (table 3).

Fusain contained a high percent of individual particles designated as rod-like in the shape analysis (table 4). The significance of this and its possible effect on coking is discussed later.

Anthracite

Three size ranges of anthracite were prepared: minus 150-mesh, minus 20-mesh, and minus 10-mesh (figs. 2, 3, and 4). Anthracite was first crushed, duplicating the established crushing procedures used in preparing the standard sample, and was divided into three equal fractions. The minus 10-mesh sample was prepared by passing one sample of the anthracite three times through the rolls set at 3 mm (1/8 inch). After each crushing the minus 10-mesh material was screened out to prevent additional crushing of the finer sizes. Roll setting of 1.3 mm (1/20 inch) was used in preparing the minus 20-mesh anthracite which was also crushed and progressively screened three times. To secure the high percentage of fine material comparable to that in the fusain, the anthracite also was hand ground and screened (table 1 and fig. 2). Although the size composition was comparable, it was not the same as that of the fusain.

Even though anthracite is the second highest rank coal recognized with a fixed carbon content of 92 to 98 percent, fusain is still discernible in it. Anthracite used in this investigation contained 7.3 percent inertinite as compared to 96.1 percent inertinite in the fusain (table 2). Anthracite, however, is a high rank coal and therefore the fixed carbon content of the sample was slightly higher and the volatile content was slightly lower than that of the fusain sample (table 3).

The shape of the individual particles was an outstanding difference between the minus 150-mesh anthracite and fusain samples (table 4). In fusain 79.6 percent of the particles were rodlike, but in the anthracite 60.9 percent of the same size material were elongate.

Coke Dust and Coke Breeze

Coke dust and coke breeze samples for this investigation were obtained from coke produced in the Survey's pilot-scale coke oven and coke testing program. The total sample for the minus 20-mesh fraction was taken from the coke dust in the tumbler test apparatus without additional preparation. The sample consisted of a relatively high percentage of minus 150- and minus 270-mesh sizes (table 1, fig. 3). Hand grinding and progressive screening was necessary to obtain the proper size distribution for the minus 150-mesh sample however. The final screen analysis of this minus 150-mesh coke dust demonstrated that its size composition was essentially the same as that of the fusain (fig. 2). Chemical analysis of the coke dust showed a lower volatile and total sulfur content and a higher fixed carbon content than those of the fusain sample (table 3). The predominate shape of the coke dust particles was elongate (table 4).

Two size fractions of the coke breeze, minus 10-mesh and 6 x 10-mesh, were screened from the sample without additional preparation. To obtain the minus 20-mesh coke breeze sample it was necessary to pass the material through the rolls set at 1/20 inch. No minus 150-mesh sample was prepared from the coke breeze. The size composition of the minus 20-mesh coke breeze and minus 20-mesh anthracite was similar (table 1 and fig. 3). Table 3 shows that chemically the coke breeze is higher in volatile and ash than the coke dust, and lower in fixed carbon. Most of the individual particles were elongate; but angular shaped particles were appreciably higher in the coke breeze than in the coke dust (table 4). In the minus 20-mesh sample of the coke breeze angular shaped particles made up 15.8 percent of the sample, whereas in the minus 20-mesh sample of coke dust angular shaped particle made up 3.8 percent of the sample.

Petroleum Coke

Petroleum coke furnished had been previously crushed, and from this material two sizes, minus 20-mesh and minus 10-mesh, were screened (table 1). The size composition of the minus 10-mesh material was roughly similar to that of the minus 10-mesh coke breeze (fig. 4). The volatile content of the petroleum coke was higher than that of fusain, anthracite, coke dust, and coke breeze, whereas the ash proved to be only 0.1 percent. The total sulfur on the moisture ash-free basis was 0.83 percent (table 3). Breakage characteristics of the petroleum coke resulted in a high percentage of elongate particles and the lowest percentages of rodlike particles (table 4).

Minus 48-Mesh Commercially Prepared Coal Dust

Minus 48-mesh commercially prepared coal dust was added to the coal charge without additional screening in hopes of improving the physical properties of coke with a minimum amount of preparation. Size composition of this sample compared more favorably with that of the minus 20-mesh coke breeze than any other sample (table 1, fig. 3). Chemically this sample was similar to samples of No. 6 and No. 5 Coals but the ash content was higher and total sulfur was lower than found in either coal (table 3).

"Fine Coal"

The minus 10-mesh, minus 20-mesh, and minus 150-mesh fractions of the standard sample of No. 6 and No. 5 Coals were obtained by first screening the standard size sample to produce the minus 10-mesh fraction. The oversize was crushed and screened for the minus 20-mesh size, and the oversize from this crushing was recrushed to produce the minus 150-mesh size consist. All three samples are referred to as "fine coal" in the tables and figures to differentiate them from the other coal samples used in this investigation. The size composition of the fine coal sample of the minus 20-mesh fraction is roughly comparable to that of the minus 20-mesh anthracite sample (table 1). Petrographic analyses showed that the maceral content of these samples is about the same as that of the other coal samples used in these tests with the exception of a slightly higher vitrinite and slightly lower exinite content in the minus 150-mesh fraction and a slightly higher percentage of inertinite in the minus 20-mesh fraction (table 2). Chemical analyses data showed only minor differences between the coal samples (table 3). The percentage of rodlike shaped particles was 51.5 for the minus 20-mesh fraction of these samples which is higher than any other additive except fusain (table 4). This may be due to a slightly higher fusain content in this size fraction.

Black Shale

Black petroliferous shale from above the No. 5 Coal was obtained from a strip mine in western Illinois. Three sizes of the shale were prepared, minus 150-mesh, minus 20-mesh, and minus 10-mesh. Breakage of black shale produced the highest amount of oversized particles in the minus 150-mesh and minus 20-mesh size compositions (table 1 and figs. 2, 3). Chemical analysis of the shale showed a high ash content, as would be expected, but also a relatively high Btu value (table 3).

"Resin" or "Asphaltene"

This light, fluffy, brown extract from coal tar pitch was obtained by dissolving coal tar pitch with boiling benzene over a steam cone and precipitating the "resin" from the filtrate with white gasoline. From each 100 grams of pitch treated 23 to 25 grams of "resin" was obtained.

A similar extract was used in an earlier investigation (Marshall et al., 1958) but petroleum ether was used instead of white gasoline. The yield was about the same in both extractions and the resulting "resin" proved beneficial in coke tests when used with the standard sample of coal and high-carbon component.

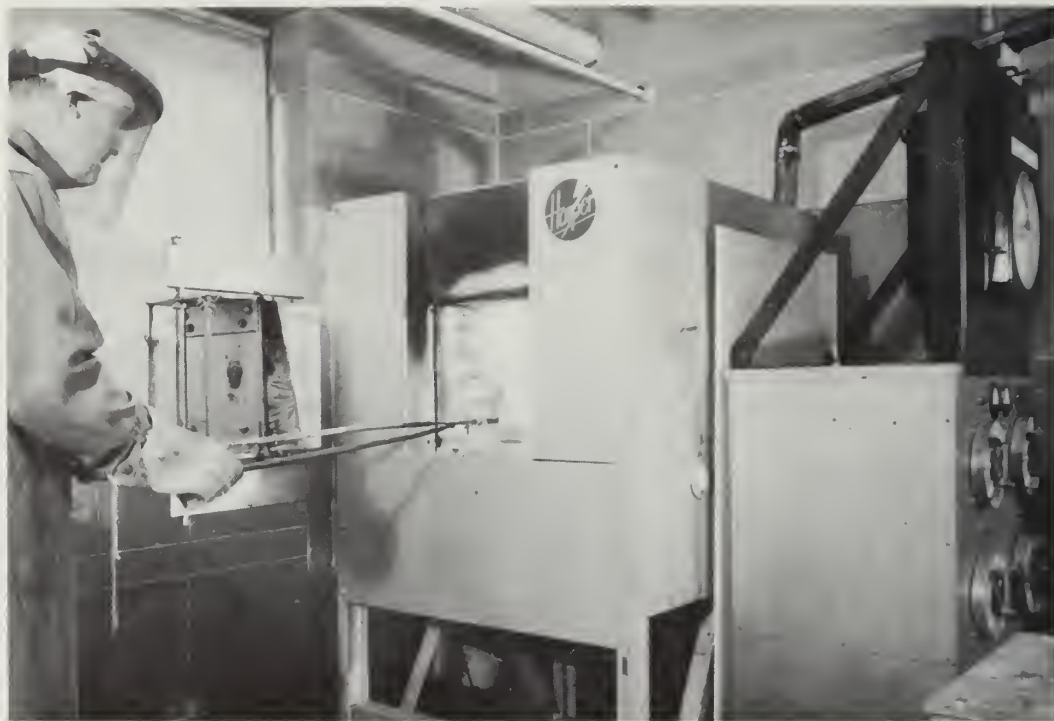


Fig. 5. Charging alumina crucibles, with fireclay cover and base, into furnace used for laboratory coking tests.

COKE PRODUCTION AND TESTING

Production

In the earlier investigation by Marshall, the furnace used for coke production, the charging temperature, rate of heating, final temperature, and final coking period were investigated and reported in detail. For the present report a brief discussion of the furnace and crucible used plus optimum coking temperatures will suffice.

The furnace is a Harper Globar type with automatic temperature controls (fig. 5). With the addition of a flue, a series of closely adjustable dampers, and an exhaust system for removal of volatiles, this furnace gave excellent service.

Crucibles used were thin-walled alumina, cylindrical, 3 inches in diameter, and approximately 6 inches in height, in which coal charges of 300 to 400 grams could be coked conveniently. Fireclay covers and bases were employed to protect both the charge and the crucibles.

Each coke run or test was made in triplicate, and because the furnace would hold nine crucibles, three different coke tests were undertaken each time the furnace was heated. The furnace was charged at 842°F (450°C), temperatures were increased at the rate of 6.5°F (3.6°C) per minute until it attained 1850°F (1010°C), at which point a constant temperature was maintained for a two-hour period. Upon removal from the furnace the contents of each crucible were quickly quenched in individual water baths, then removed from the water, placed in individual pans, and dried for two days on the furnace top.

Testing

Several methods of coke testing on the laboratory scale were developed in the previous study by Marshall, two of which (ASTM shatter and tumbler tests modified to laboratory scale), were employed in the present investigation. Preliminary studies have indicated that there is a relationship between the physical properties of laboratory test coke and pilot plant cokes, although the values of shatter and tumbler indices differ in magnitude.

Because tumbler tests probably reflect quality of coke more than shatter tests, in the present study two of the three samples of each coke run were subjected to tumbler tests, and the tumbler indices given are the average of these two tests; the third sample was used for shatter tests. Stability indices were determined by the percentage of material retained on the 1-inch screen, and resistance to abrasion (hardness) was determined by the percentage of material retained on the 1/4-inch screen. For this investigation the minus 1/4-inch index was plotted. The plus 1-inch fraction from the shatter tests was taken as the shatter index. Percentage shatter data also have been plotted on plus 1/4 and minus 1/4-inch fractions.

It is realized that the modified shatter and tumbler tests used in this investigation, like the standard ASTM tests, are highly empirical and small differences between test results may not be significant.

COKING STUDY RESULTS

Effect of Blending No. 6 and No. 5 Coals in Various Amounts

To establish a standard sample with which various sizes and amounts of additives were to be combined, Illinois No. 6 and No. 5 Coals of the basic size composition were mixed and coked. Seven mixtures of the two coals were used and the results of the tests are given in table 5 and figure 6. Coal mixtures in this series varied, in 10 percent increments, from a blend of 90 percent No. 6 and 10 percent No. 5 Coal to 30 percent No. 6 and 70 percent No. 5 Coal. One run of 100 percent No. 6 Coal also was made. In evaluating coke data, although tumbler indices were considered more significant than shatter indices, shatter indices were not disregarded. When the tumbler indices of two cokes were essentially equal, the shatter indices were utilized as the deciding factor in evaluating which coke was better.

Variations were not great in the shatter and tumbler indices of cokes produced from the controlled blends of No. 6 and No. 5 Coals but they were erratic, (table 5, fig. 6). Maximum values for all shatter and tumbler indices were not found in any one coal blend tested, therefore it was necessary to choose a blend which gave optimum over-all values after the importance of each index had been considered and evaluated. Shatter minus 1/4-inch showed little change through the test series. Shatter plus 1-inch reached a maximum in the blend with 90 percent No. 6 Coal, but the tumbler indices showed little if any improvement.

The tumbler minus 1/4-inch showed a slight improvement in the 60 percent No. 6 Coal blend, and the tumbler plus 1-inch reached its maximum value in the blend with 70 percent No. 6 Coal. Duplicate tests of the blend containing 70 percent No. 6 Coal substantiated this high tumbler plus 1-inch index, and other indices show a relatively stable or slightly improved value. Therefore the 70 percent No. 6 Coal blend was chosen as the standard sample for all subsequent tests of this investigation.

Table 5. - Character of Cokes Produced from Standard Samples
of No. 6 and No. 5 Coals with Various Additives
Charging temperature 850°F; final temperature 1850°F

Run no.	Coal No. 6 %	Coal No. 5 %	Additive	%	Shatter			Tumbler		
					+ 1"	+1/4"	-1/4"	+ 1"	+1/4"	-1/4"
1A	100.0	0.0			93.0	96.8	3.2	80.5	86.6	13.4
1B	70.0	30.0			91.0	96.5	3.5	87.5	87.1	12.9
1C	60.0	40.0			88.6	96.1	3.9	83.0	88.6	11.4
2D	50.0	50.0			88.2	96.2	3.8	83.8	87.7	12.3
2E	40.0	60.0			91.6	96.0	4.0	80.8	90.5	9.5
2F	30.0	70.0			89.5	96.1	3.9	82.8	88.2	11.8
3A	80.0	20.0			92.6	96.7	3.3	75.0	88.8	11.2
3B	90.0	10.0			95.3	97.0	3.0	80.5	88.2	11.8
3C	70.0	30.0			94.2	96.5	3.5	84.2	88.6	11.4
4D	66.5	28.5	Fusain -150 mesh	5	95.1	95.6	4.3	81.0	85.6	14.4
4E	63.0	27.0	Fusain -150 mesh	10	92.7	94.0	6.0	90.2	90.6	9.4
4F	59.5	25.5	Fusain -150 mesh	15	83.9	88.8	11.2	80.3	80.4	19.6
5A	56.0	24.0	Fusain -150 mesh	20	82.3	87.4	12.6	68.4	71.1	28.9
5B	66.5	28.5	Coke dust -150 mesh	5	95.6	96.0	4.0	87.4	87.9	12.1
5C	63.0	27.0	Coke dust -150 mesh	10	85.9	92.9	7.1	81.5	85.6	14.4
6D	59.5	25.5	Coke dust -150 mesh	15	87.5	90.9	9.1	82.5	84.9	15.1
6E	56.0	24.0	Coke dust -150 mesh	20	84.6	89.2	10.8	62.8	67.5	32.5
6F	66.5	28.5	Coke dust -20 mesh	5	91.3	95.9	4.1	87.1	87.2	12.8
7A	63.0	27.0	Coke dust -20 mesh	10	87.7	94.5	5.5	90.4	91.7	8.3
7B	59.5	25.5	Coke dust -20 mesh	15	84.8	93.4	6.6	88.4	88.4	11.6
7C	56.0	24.0	Coke dust -20 mesh	20	83.3	89.0	11.0	80.6	80.6	19.4
8D	66.5	28.5	Coke breeze -10 mesh	5	91.3	94.6	5.4	86.4	86.5	13.5
8E	63.0	27.0	Coke breeze -10 mesh	10	82.3	92.7	7.3	79.9	84.0	16.0
8F	59.5	25.5	Coke breeze -10 mesh	15	67.8	83.1	16.9	58.4	58.4	41.6
9A	56.0	24.0	Coke breeze -10 mesh	20	63.9	76.5	23.5	24.3	30.1	69.9
9B	66.5	28.5	Coke breeze 6x10 mesh	5	60.4	90.5	9.5	40.0	75.2	24.8
9C	63.0	27.0	Coke breeze 6x10 mesh	10	47.6	79.8	20.2	16.0	55.1	44.9
10D	59.5	25.5	Coke breeze 6x10 mesh	15	21.7	67.9	32.1	3.9	39.6	60.4
10E	56.0	24.0	Coke breeze 6x10 mesh	20	13.4	54.5	45.5	4.9	29.2	70.8
10F	66.5	28.5	Anthracite -150 mesh	5	87.7	94.2	5.8	79.2	83.8	16.2
11A	63.0	27.0	Anthracite -150 mesh	10	91.8	92.4	7.6	82.5	85.3	14.7
11B	59.5	25.5	Anthracite -150 mesh	15	85.7	88.8	11.2	77.4	79.1	20.9
11C	56.0	24.0	Anthracite -150 mesh	20	32.3	50.0	50.0	39.9	43.8	56.2
12D	66.5	28.5	Anthracite -20 mesh	5	86.1	96.2	3.8	89.3	90.6	9.4
12E	63.0	27.0	Anthracite -20 mesh	10	89.3	96.0	4.0	90.6	91.6	8.4
12F	59.5	25.5	Anthracite -20 mesh	15	87.0	94.1	5.9	83.7	87.3	12.7
13A	56.0	24.0	Anthracite -20 mesh	20	87.1	94.7	5.3	86.6	87.3	12.7
13B	66.5	28.5	Anthracite -10 mesh	5	88.0	95.9	4.1	85.8	89.2	10.8
13C	63.0	27.0	Anthracite -10 mesh	10	91.7	96.0	4.0	88.2	89.9	10.1
14D	59.5	25.5	Anthracite -10 mesh	15	91.2	95.8	4.2	84.7	87.9	12.1
14E	56.0	24.0	Anthracite -10 mesh	20	87.5	94.7	5.3	87.3	89.4	10.6
14F	66.5	28.5	Petroleum coke -20 mesh	5	89.2	95.9	4.1	82.8	85.9	14.1

Table 5. - Continued

Run No.	Coal No. 6 %	Coal No. 5 %	Additive	%	Shatter			Tumbler		
					+1"	+1/4"	-1/4"	+1"	+1/4"	-1/4"
15A	63.0	27.0	Petroleum coke -20 mesh	10	88.2	96.0	4.0	79.5	89.5	10.5
15B	59.5	25.5	Petroleum coke -20 mesh	15	92.9	96.2	3.8	87.4	87.5	12.5
15C	56.0	24.0	Petroleum coke -20 mesh	20	87.3	94.3	5.7	85.4	85.6	14.4
16D	66.5	28.5	Petroleum coke -10 mesh	5	86.7	95.8	4.2	88.3	89.0	11.0
16E	63.0	27.0	Petroleum coke -10 mesh	10	92.6	95.6	4.4	81.0	87.0	13.0
16F	59.5	25.5	Petroleum coke -10 mesh	15	94.2	95.5	4.5	78.5	84.6	15.4
17A	56.0	24.0	Petroleum coke -10 mesh	20	92.6	95.9	4.1	81.5	87.4	12.6
17B	66.5	28.5	"Fine coal" -10 mesh	5	94.3	96.9	3.1	79.9	87.8	12.2
17C	63.0	27.0	"Fine coal" -10 mesh	10	88.3	96.3	3.7	79.2	87.7	12.3
18D	59.5	25.5	"Fine coal" -10 mesh	15	95.5	96.1	3.9	82.3	87.4	12.6
18E	56.0	24.0	"Fine coal" -10 mesh	20	87.0	95.8	4.2	82.8	89.0	11.0
18F	66.5	28.5	"Fine coal" -150 mesh	5	95.9	96.3	3.7	84.4	88.0	12.0
19A	63.0	27.0	"Fine coal" -150 mesh	10	86.2	96.3	3.7	77.4	88.4	11.6
19B	59.5	25.5	"Fine coal" -150 mesh	15	92.9	96.3	3.7	82.6	86.4	13.6
19C	56.0	24.0	"Fine coal" -150 mesh	20	96.1	96.3	3.7	77.9	83.0	17.0
20D	66.5	28.5	"Fine coal" -20 mesh	5	95.2	95.3	4.7	89.9	90.1	9.9
20E	63.0	27.0	"Fine coal" -20 mesh	10	90.1	95.2	4.8	86.1	88.9	11.1
20F	59.5	25.5	"Fine coal" -20 mesh	15	86.4	93.3	6.7	82.1	84.2	15.8
21A	66.5	28.5	Black shale -20 mesh	5	89.2	96.2	3.8	85.8	90.4	9.6
21B	63.0	27.0	Black shale -20 mesh	10	83.0	95.2	4.8	83.7	89.4	10.6
21C	59.5	25.5	Black shale -20 mesh	15	85.1	91.6	8.4	75.8	78.4	21.6
22D	68.25	29.25	Black shale -20 mesh	2 $\frac{1}{2}$	90.0	95.8	4.2	83.5	86.6	13.4
22E	64.75	27.75	Black shale -20 mesh	7 $\frac{1}{2}$	91.2	95.5	4.5	80.2	86.4	13.6
22F	68.25	29.25	Black shale -10 mesh	2 $\frac{1}{2}$	89.7	94.2	5.8	78.8	83.8	16.2
23A	66.5	28.5	Black shale -10 mesh	5	87.3	94.9	5.1	79.7	86.5	13.5
23B	64.75	27.75	Black shale -10 mesh	7 $\frac{1}{2}$	86.9	94.8	5.2	80.7	86.0	14.0
23C	63.0	27.0	Black shale -10 mesh	10	89.2	92.9	7.1	77.9	80.9	19.1
24D	66.5	28.5	Fusain 20 x 150 mesh	5	89.6	94.5	5.5	84.8	89.3	10.7
24E	63.0	27.0	Fusain 20 x 150 mesh	10	86.5	92.2	7.8	85.5	87.1	12.9
24F	59.5	25.5	Fusain 20 x 150 mesh	15	75.6	84.3	15.7	69.4	69.6	30.4
25A	70.0	30.0	"Resin"	5	95.8	97.2	2.8	89.0	89.9	10.1
25B	63.0	27.0	Fusain -150 mesh "Resin"	10 5	91.6	96.0	4.0	93.5	93.7	6.3
25C	56.0	24.0	Fusain -150 mesh "Resin"	20 5	85.4	86.4	13.6	70.8	70.8	29.2
26D	49.0	21.0	Fusain -150 mesh "Resin"	30 5	76.4	77.7	22.3	39.2	39.2	60.8

* Standard sample of 70 percent No. 6 Coal and 30 percent No. 5 Coal reduced to finer sizes.

Table 5. - Continued

Run no.	Coal No. 6 %	Coal No. 5 %	Additive	%	Shatter			Tumbler			
					+1"	+1/4"	-1/4"	+1"	+1/4"	-1/4"	
26E	63.0	27.0	Anthracite -20 mesh "Resin"	10 5	87.6	96.3	3.7	85.1	87.8	12.2	
26F	56.0	24.0	Anthracite -20 mesh "Resin"	20 5	95.7	96.6	3.4	91.7	91.7	8.3	
27A	59.5	25.5	Coke dust -20 mesh Petroleum coke	10 5	90.3	92.3	7.7	85.7	85.7	14.3	
27B	52.8	22.5	Coke dust -20 mesh Petroleum coke	15 10	87.2	89.6	10.4	78.7	78.9	21.1	
27C	56.0	24.0	Coke dust -20 mesh Petroleum coke	10 10	81.7	88.4	11.6	70.9	71.2	28.8	
28A	66.5	28.5	Coke breeze -20 mesh	5	89.1	95.2	4.8	81.6	89.4	12.6	
28B	63.0	27.0	Coke breeze -20 mesh	10	93.0	94.8	5.2	85.2	85.4	14.6	
28C	59.5	25.5	Coke breeze -20 mesh	15	82.3	86.8	13.2	54.7	54.9	45.1	
29D	66.5	28.5	Black shale -150 mesh	5	86.4	95.6	4.4	71.5	84.3	15.7	
29E	63.0	27.0	Black shale -150 mesh	10	88.6	94.9	5.1	73.3	79.5	20.5	
29F	59.5	25.5	Black shale -150 mesh	15	81.0	86.1	13.9	48.1	58.0	42.0	
30A	63.0	27.0	Petroleum coke -10 mesh "Resin"	10 5	96.9	97.1	2.9	75.3	86.2	13.8	
30B	56.0	24.0	Petroleum coke -10 mesh "Resin"	20 5	94.8	96.9	3.1	85.0	87.5	12.5	
30C	59.5	25.5	Petroleum coke -10 mesh Coke dust -20 mesh	10 5	88.3	94.9	5.1	86.3	86.4	13.6	
31D	63.0	27.0	Coke dust -20 mesh "Resin"	10 5	93.1	95.8	4.2	83.8	86.2	13.8	
*31E	56.0	24.0	Coke dust -20 mesh "Resin"	20 5	90.0	91.0	9.0	78.1	78.1	21.9	
31F	63.0	27.0	Fusain -150 mesh Coal tar pitch	10 5	93.9	96.0	4.0	87.0	88.6	11.4	
32A	66.5	28.5	Coal dust -48 mesh	5	91.1	96.8	3.2	86.4	87.7	12.3	
32B	63.0	27.0	Coal dust -48 mesh	10	86.7	96.4	3.6	81.8	86.6	13.4	
32C	59.5	25.5	Coal dust -48 mesh	15	87.6	93.1	6.9	78.2	82.4	17.6	
3	Average of Run 1B and 3C - Basic blend for blending with additives.					92.2	96.5	3.5	85.2	88.1	11.9

* One charge lost in the oven - 1 tumbler test only.

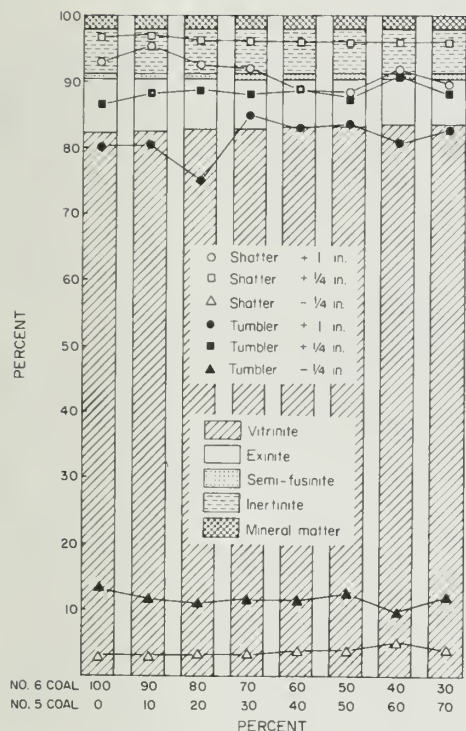


Fig. 6. Petrographic analysis and character of coke produced from different blends of No. 6 and No. 5 Coals of basic size composition.

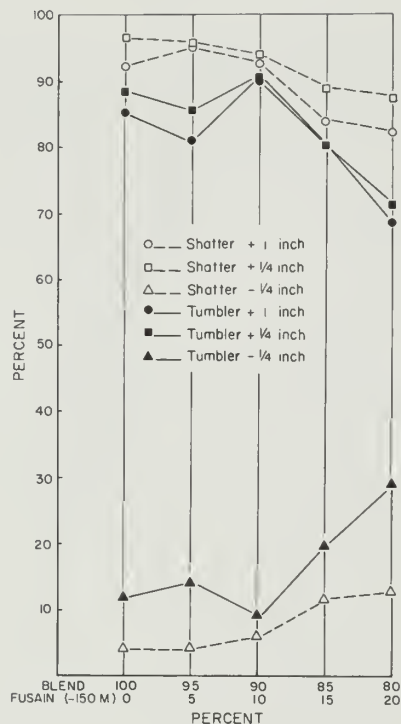


Fig. 7. FUSAIN - Character of coke produced by blending different amounts of minus 150-mesh fusain with the standard sample.

Effect of Fusain Additive

Recent laboratory studies (Marshall et al., 1958) and limited pilot-scale studies (Harrison, 1958) have indicated that addition of fusain in an optimum amount to the coals tested generally improves the physical properties of the resulting coke. In the present investigation 5, 10, 15, and 20 percent minus 150-mesh fusain was mixed with the standard sample of No. 6 and No. 5 Coals. Because natural breakage of relatively pure fusain resulted in a size fraction of minus 150-mesh material, it was impossible to obtain fusain of a larger size composition that was not heavily mineralized. Coke tests were made using the 20 x 150-mesh fusain, but the marked increase in visible mineral matter made this size fraction unsuitable (table 2). The petrographic analysis shows that the pyrite content of the 20 x 150-mesh fusain was 10.8 percent. The higher percentage of mineral matter also probably introduces other factors that affect the coke characteristics besides those of the coal components, but the other factors were not evaluated in this investigation.

For undetermined reasons the tumbler plus 1-inch index of coke, produced by addition of 5 percent minus 150-mesh fusain, decreased below the tumbler plus 1-inch index of the coke produced from the standard sample (fig. 7), as it had in earlier investigations by Marshall. Addition of 10 percent minus 150-mesh fusain

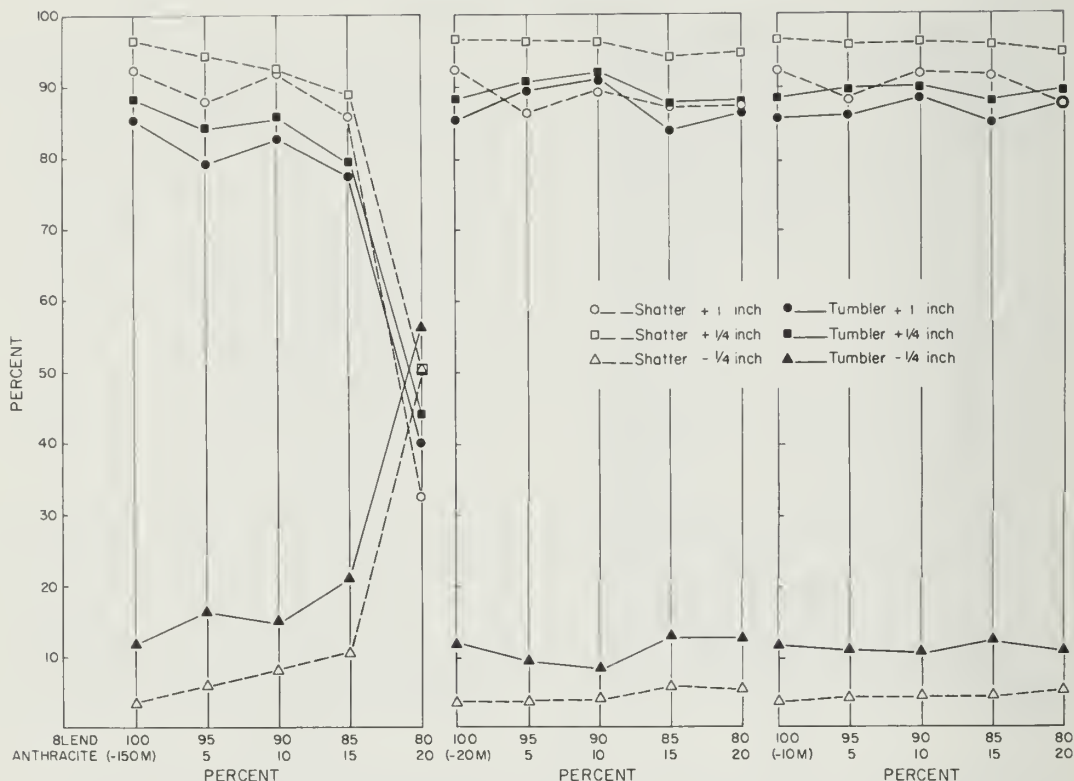


Fig. 8. ANTHRACITE - Character of coke produced by blending minus 150-mesh, minus 20-mesh, and minus 10-mesh anthracite with the standard sample.

to the coal charge, however, produced the high of 90.2 for the tumbler plus 1-inch index. The tumbler plus 1/4-inch index responded accordingly with 90.6.

Shatter plus 1-inch index rose to 95.1 with the addition of 5 percent minus 150-mesh fusain and declined thereafter, but was still higher than the standard sample results with the addition of 10 percent fusain. Fusain has no fusion property of its own, hence, when an excessive amount is present which cannot be incorporated by the vitrinite and other macerals in the softening period of carbonization, weak areas occur within the coke. This was strikingly demonstrated when 15 percent fusain was added to the coal charge and all coke indices showed a decline (fig. 7) and continued to decline with the addition of 20 percent fusain.

Effect of Anthracite Additive

Blending anthracite with coal for production of metallurgical coke is not new but because results vary, the effect of amount and size of the anthracite blended with Illinois coals was studied. The results obtained apply only to the anthracite used, therefore variations between anthracites may have differing effects upon the cokes produced. As previously stated, three size samples of anthracite were prepared - minus 150-mesh, minus 20-mesh, and minus 10-mesh. Each size fraction was blended with the standard sample in amounts of 5, 10, 15, and 20 percent and the results are shown in table 5 and figure 8.

Addition of minus 150-mesh anthracite to the standard sample produced cokes whose shatter and tumbler indices were inferior to those of the standard sample (table 5, fig. 8). It was noted that the addition of 5 percent minus 150-mesh anthracite had an effect similar to that of adding the same amount of fusain. The tumbler plus 1-inch and plus 1/4-inch as well as the shatter plus 1-inch indices indicated a coke inferior to that produced from the standard sample alone. The blending of 10 percent minus 150-mesh anthracite produced an improvement over the 5 percent blend, but not to the same extent as that with the fusain blend. Addition of 15 to 20 percent minus 150-mesh anthracite resulted in cokes whose indices were greatly decreased.

The effect on the coke values of blending minus 20-mesh samples of anthracite with the standard sample of coal was decidedly different from those produced by blending the minus 150-mesh anthracite and fusain (table 5, fig. 8). All tumbler indices improved when blended with 5 percent minus 20-mesh anthracite. Addition of 10 percent minus 20-mesh anthracite produced one of the best cokes made from any blend which included only the standard sample and a high-carbon component. The tumbler plus 1-inch index rose to 90.6 and the tumbler plus 1/4-inch index rose to 91.6. The tumbler plus 1-inch index dropped to 83.7 with addition of 15 percent minus 20-mesh anthracite, but instead of continuing to decline as in previous tests with minus 150-mesh fusain and anthracite, this index rose to 86.6 with addition of 20 percent minus 20-mesh anthracite. The tumbler plus 1/4-inch index declined and all shatter indices declined with the addition of 15 percent minus 20-mesh anthracite. Addition of 20 percent minus 20-mesh anthracite produced only minor changes in the tumbler plus 1/4-inch index and in the shatter indices.

Shatter and tumbler indices determined on cokes produced from blends of minus 10-mesh anthracite gave results similar to those produced from blends of minus 20-mesh anthracite (fig. 8). Optimum results for this particular additive were obtained with the blending of 10 percent minus 10-mesh anthracite, but the amount of improvement in the tumbler plus 1-inch or plus 1/4-inch was not as great as corresponding values obtained with the minus 20-mesh anthracite.

For the anthracite tested the optimum size for addition to the standard sample proved to be minus 20-mesh and the optimum amount was 10 percent.

Effect of Coke Dust Additive

Improved shatter and tumbler plus 1-inch indices resulted from blending 5 percent minus 150-mesh coke dust with the standard sample which is opposite to the effect produced by adding 5 percent minus 150-mesh fusain or anthracite to the standard sample (table 5 and fig. 9). Addition of 10 percent minus 150-mesh coke dust produced a decline in all indices. Slight variations were found with the addition of 15 percent of this material, but 20 percent gave cokes of poor physical properties. Shatter and tumbler 1/4-inch indices decreased throughout the range of this series of tests.

Optimum results were obtained with coke dust, as they had been with anthracite, by blending the minus 20-mesh material with the standard sample (table 5, fig. 9). An increase in the plus 1-inch tumbler index was attained in coke produced by blending 5 percent minus 20-mesh coke dust with the standard sample; all other indices were constant or declined from the values obtained from only the standard sample. Tumbler indices increased, but shatter indices demonstrated a downward trend when 10 percent minus 20-mesh coke dust was used in the blend, nevertheless they remained relatively high until 15 percent of this material was

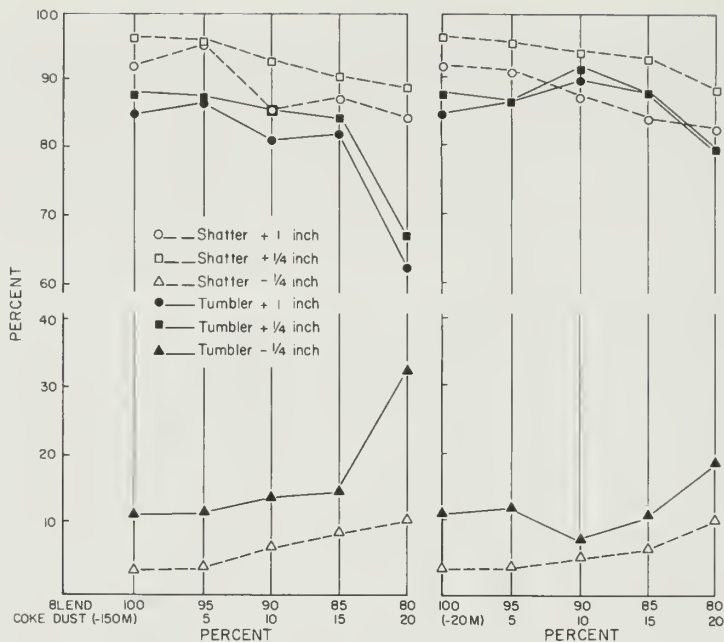


Fig. 9. COKE DUST - Character of coke produced by blending minus 150-mesh and minus 20-mesh coke dust with the standard sample.

blended with the standard sample. Coke dust minus 20-mesh and anthracite minus 20-mesh gave essentially the same results in the 10 percent additive blend (table 5, figs. 8, 9).

Effect of Coke Breeze Additive

Although the chemical composition of the coke breeze and the coke dust are similar (table 3), the character of coke produced by mixing these additives with the standard sample varied a great deal as shown by the shatter and tumbler indices (table 5, figs. 9, 10).

The physical properties of cokes produced by adding 5 and 10 percent minus 20-mesh coke breeze remained about the same as the physical property of cokes produced from the standard sample. Addition of 15 percent minus 20-mesh coke breeze produced a decline in all coke properties.

The only coke-breeze blend in this series that gave cokes that might be considered as good as those produced from the standard sample was the one that contained 5 percent minus 10-mesh coke breeze (fig. 10).

Drastic deterioration in coke character was produced by mixing the standard sample with 6 x 10-mesh coke breeze. Although the coke remained in one piece upon being discharged from the crucible, and shrinkage appeared to be slight, the tumbler plus 1-inch index was only 40 and the shatter plus 1-inch index was only 60.4. Blending 10 percent 6 x 10-mesh coke breeze caused a further drop in tumbler plus 1-inch to 16 and in the shatter plus 1-inch index to 47.6 (table 5, fig. 10).

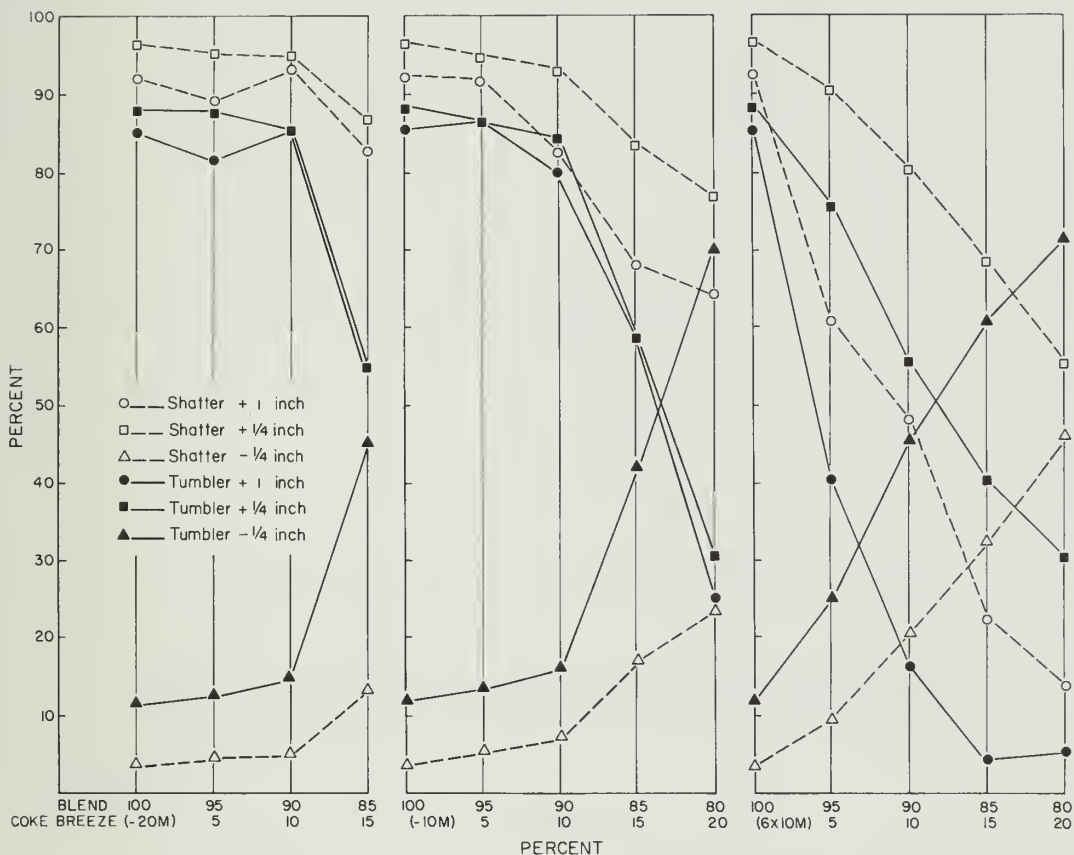


Fig. 10. COKE BREEZE - Character of coke produced by blending minus 20-mesh, minus 10-mesh, and 6 x 10-mesh coke breeze with the standard sample.

Effect of Petroleum Coke Additive

Generally maximum values for both tumbler plus 1-inch and tumbler plus 1/4-inch were attained by blending some definite amount of an additive with the standard sample. This was not true, however, in the minus 20-mesh fraction of the petroleum coke additive (table 5, fig. 11). Fifteen percent minus 20-mesh petroleum coke was necessary to give maximum tumbler plus 1-inch index, but only 10 percent minus 20-mesh petroleum coke was needed to give the maximum tumbler plus 1/4-inch index.

For this series of laboratory tests, data showed that 15 percent minus 20-mesh petroleum coke is needed to give optimum shatter and tumbler plus 1-inch indices. Generally 10 percent is an adequate amount for other additives used in this investigation.

A blend containing 5 percent minus 10-mesh petroleum coke plus the standard sample produced a coke of optimum physical properties in this series. All indices remained relatively high in the cokes produced from petroleum coke blends, both minus 10-mesh and minus 20-mesh (fig. 11), and did not show the large decline observed with the coke breeze additive.

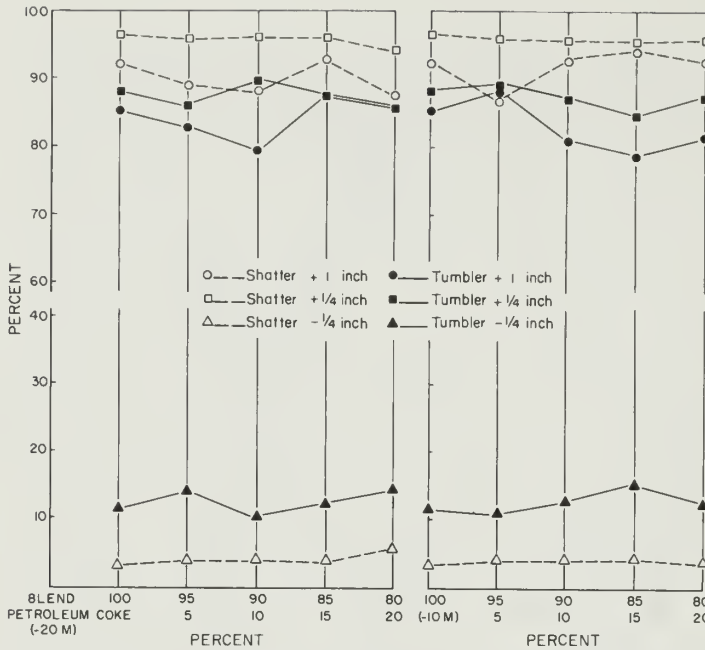


Fig. 11. PETROLEUM COKE - Character of coke produced by blending minus 20-mesh and minus 10-mesh petroleum coke with the standard sample.

Effect of "Fine Coal" Additive

To compare the relative effect on coke character of reduced sizes of the coal itself and the relatively high-carbon additives, a sample of the 70 percent No. 6 Coal - 30 percent No. 5 Coal was reduced to sizes comparable to those of the high-carbon components and substituted for them in coal blends for laboratory tests. Physical properties of cokes obtained from blending fine coal with the standard sample are shown in figure 12. Five percent minus 20-mesh "fine coal" produced a coke having indices that showed improvement over the coke produced from the standard sample. Table 5 shows that this coke compares fairly well with the coke produced by using 10 percent minus 150-mesh fusain.

Effect of Black Shale Additive

In order to evaluate the influence of black shale as an additive in the coking process, three sizes of shale were prepared and blended in various amounts with the coal. As the shale contained a higher ash content than other additives, 2.5 and 7.5 percent shale blends were included in the series when testing the minus 20-mesh and minus 10-mesh shales (table 5, fig. 13).

Resulting shatter and tumbler indices disclosed there was no improvement from using the black shale additive except for a slight increase in tumbler indices with 5 percent minus 20-mesh shale in the blend. Shatter and tumbler indices of cokes decreased rapidly as higher percentages of minus 150-mesh shale were added. Results were erratic as minus 20-mesh shale was added, with the slight improvement in the blend that contained 5 percent shale, but no rapid decrease in indices

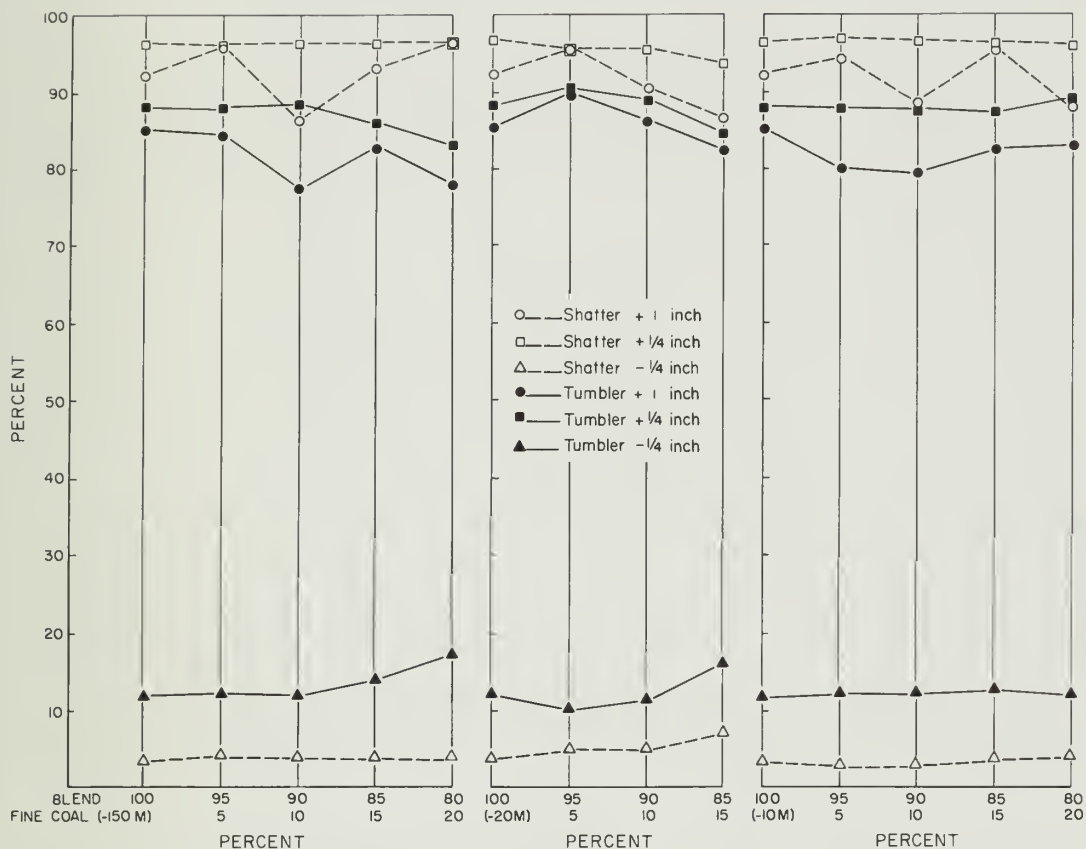


Fig. 12. "FINE COAL" - Character of coke produced by blending reduced sizes of the standard sample (minus 150-mesh, minus 20-mesh, and minus 10-mesh) with the standard sample.

was found with the addition of up to 15 percent shale (fig. 13). The minus 10-mesh size produced an initial decline in all indices, after which there was a leveling off through the 10 percent addition of shale.

Recent investigations in Australia have indicated that a bentonitic shale additive improved the coking properties of a local coal (Gregory and Felton, 1959). Considering this fact it is suggested that additional laboratory tests are desirable in which not only size, amount, shape, and chemical analysis are considered, but the mineral composition of the shale should be thoroughly investigated.

Effect of Minus 48-mesh Coal

As stated previously it was hoped that the minus 48-mesh "coal dust" from preparation plants could be utilized without additional preparation. Unfortunately, petrographic analyses of the material sampled exhibited an inertinite (including fusain) content of only 13.2 percent (table 2). Additional screening through a 150-mesh screen showed that the inertinite content could be increased to about 18.6 percent, but only 27.3 percent of the total sample was smaller than 150-mesh.

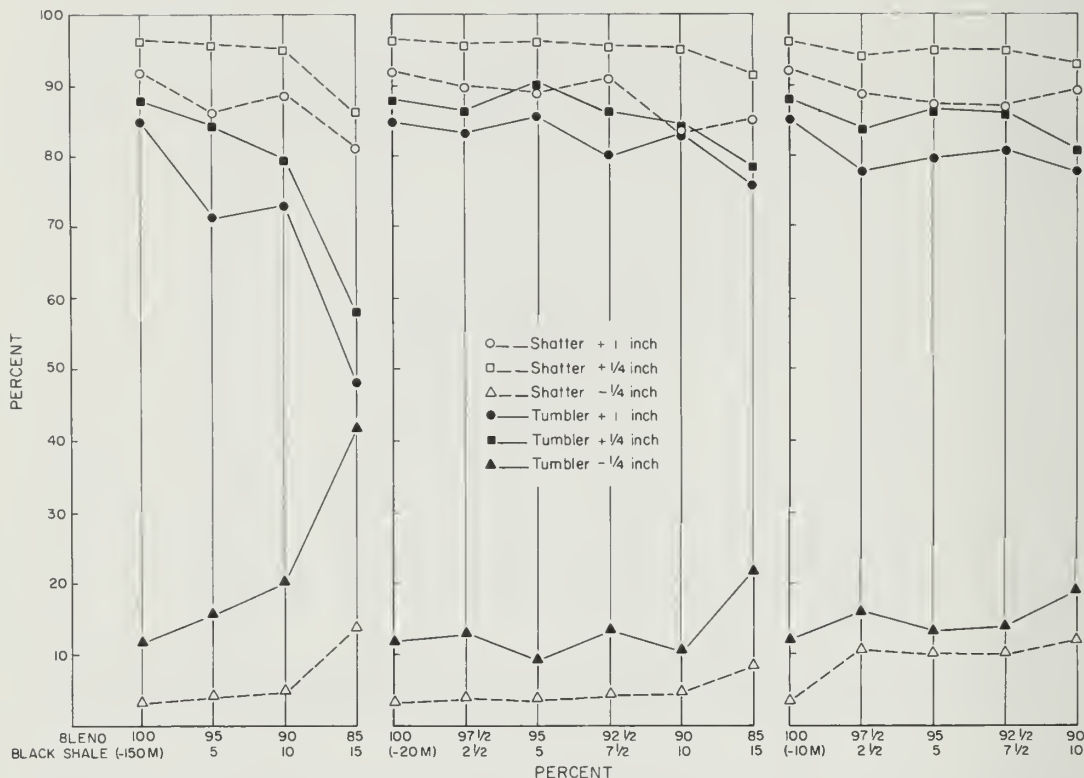


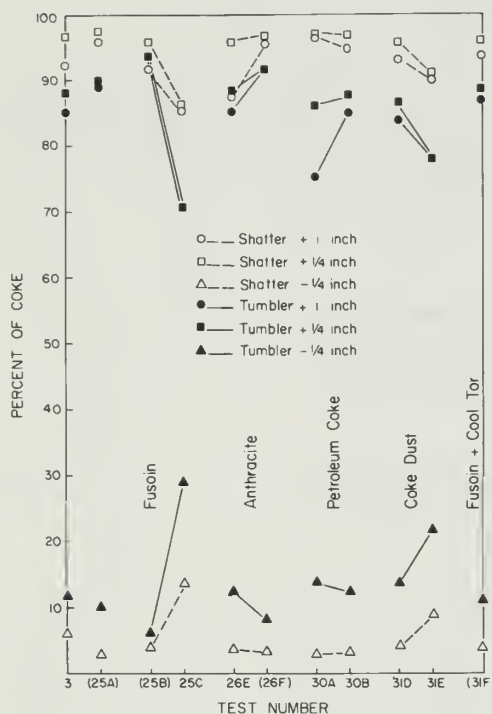
Fig. 13. BLACK SHALE - Character of coke produced by blending minus 150-mesh, minus 20-mesh, and minus 10-mesh black shale with the standard sample.

Cokes produced by blending minus 48-mesh coal with the standard sample showed no improvement except for the 5 percent blend. In this coke the indices for tumbler plus 1-inch showed a possible improvement (table 5). Although this trend is slight it warrants additional consideration in future testing, as this material possibly could be a source of a high-fusain material.

An earlier report (Thiessen, 1936) stated that dedusting plant dust was composed of 41.4 percent fusain and that 86 percent of this fusain is contained in the minus 200-mesh fraction. Although this was not found true in a limited investigation of coal dust from a modern preparation plant, it is possible that changes in the methods of mining may be responsible for this variation. The continuous miners tend to grind up all coal macerals, so it is probable that the high percentage of fusain that was found in earlier investigations in the fine coal may now be diluted with the fine vitrinite and exinite.

Effect of "Resin" or "Asphaltene"

The previous study by Marshall et al., (1958) demonstrated that a benzene-soluble, petroleum ether insoluble, extract of coal tar pitch (termed "resin" or "asphaltene"), when blended with a standard sample of coal and fusain, markedly improved the quality of laboratory coke produced. For the present investigation a "resin" was used that was similar to that of the previous tests except that white gasoline was used instead of petroleum ether to precipitate the final product.



TEST NUMBER	COAL NO. 6	PERCENT NO. 5	ADDITIVES	PERCENT	OTHERS	PERCENT
3	70	30				
25A	70	30			"Resin"	5
25B	63	27	Fusain (-150M)	10	"Resin"	5
25C	56	24	Fusain (-150M)	20	"Resin"	5
26E	63	27	Anthracite (-20M)	10	"Resin"	5
26F	56	24	Anthracite (-20M)	20	"Resin"	5
30A	63	27	Petroleum Coke (-10M)	10	"Resin"	5
30B	56	24	Petroleum Coke (-10M)	20	"Resin"	5
31D	63	27	Coke Dust (-20M)	10	"Resin"	5
31E	56	24	Coke Dust (-20M)	20	"Resin"	5
31F	63	27	Fusain (-150M)	10	Coal Tar	5

Fig. 14. "RESIN" - Character of coke produced by blending 5 percent "resin" with the standard sample (25A) and various amounts of different high carbon components. In test 31F coal tar pitch was used instead of "resin." Test 3, which shows results produced by coking the standard sample alone, is included for comparison. Test numbers enclosed in parentheses represent cokes that were better than the coke in test 3.

Laboratory coking tests using "resins" from both types of preparation gave results that were essentially the same.

"Resin" was blended with those higher carbon components in which mixtures of additives and standard sample of coal gave the optimum or best results for each additive used (table 5). The one exception was coke breeze. For blends not containing "resin," calculations were made so that the standard sample plus the additive equaled 100 percent. For the blends containing "resin" the same calculations were used and 5 percent additional "resin" was blended into this mixture. Results of coke tests are shown in figure 14. Parentheses around the test number indicates that the coke quality of that test is better than coke quality produced from the standard sample (as determined by shatter and tumbler indices).

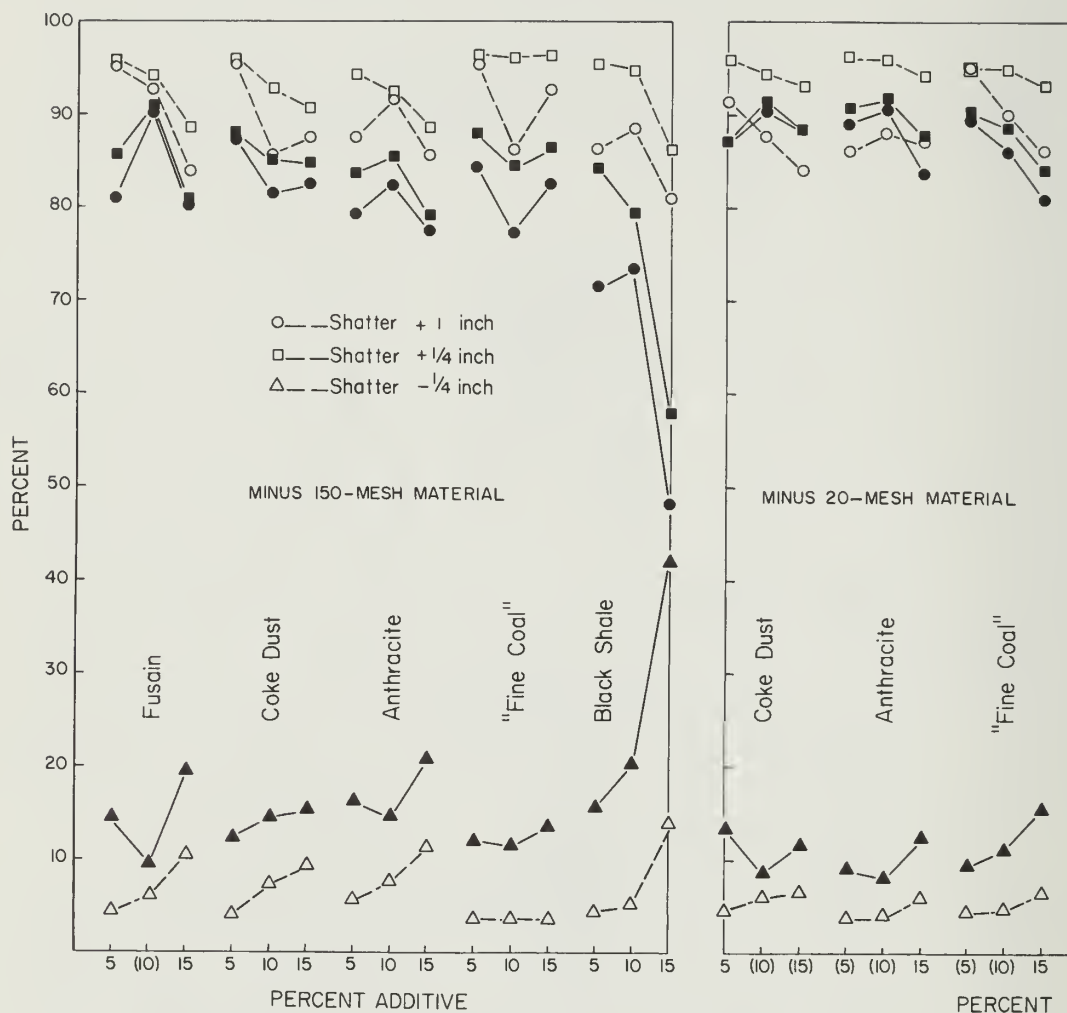
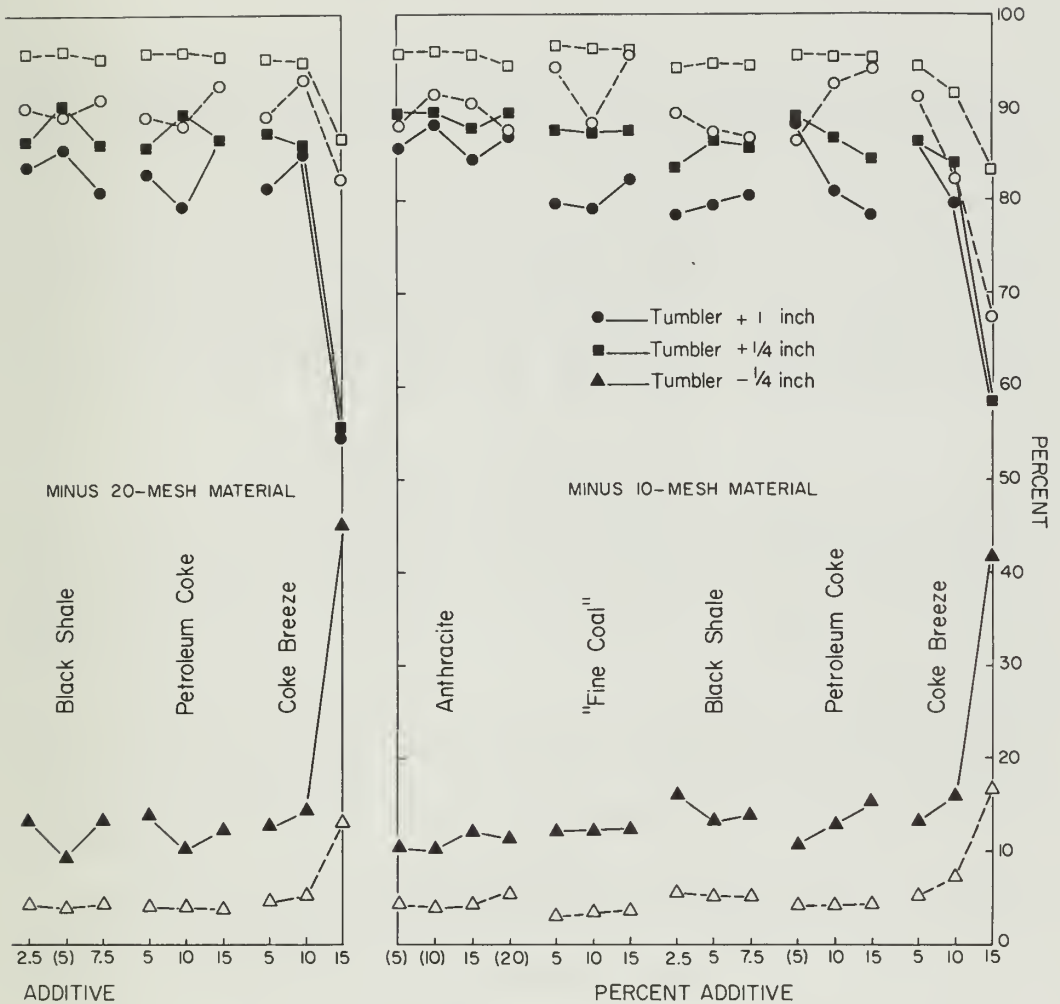


Fig. 15. Character of coke showing effect of size composition, nature,

Test 25A (fig. 14) demonstrated that coke quality is improved by addition of "resin" to the standard sample (test 3) except for the shatter plus 1-inch which remained about the same. Addition of "resin" to the blend containing 10 percent fusain produced an excellent coke (test 25B), but addition of "resin" to the blend containing 20 percent fusain resulted in a coke in which all coke indices deteriorated (test 25C).

Data obtained from the anthracite blends indicated that 20 percent minus 20-mesh anthracite with "resin" produced a coke with considerably improved characteristics. Neither petroleum coke nor coke dust reacted favorably with "resin" for the production of an improved coke.

One test was made using as an additive coal tar pitch which was the same material from which the resin was derived. Indications from this one test are that a blend (test No. 31F, fig. 14) containing the standard sample, 10 percent fusain, and this pitch will produce a coke not significantly better than from the standard sample alone (test 3 and fig. 14), and not as good a coke as the standard sample and 10 percent fusain alone (table 5).



and amount of additive blended with the standard sample.

FACTORS WHICH POSSIBLY AFFECT COKING RESULTS WHEN ADDITIVES ARE USED

Numerous factors are responsible for results obtained in the various series of laboratory scale coking tests, some of which are known and some unknown. Standardization of certain variables such as charging temperature, rate of heating, final coking temperature, final soaking temperature, and standard method of preparing the coal charge have aided in evaluating some of the other factors. In this investigation four variables have been considered; the amount of the additive, its particle size, the nature of the additive, and the shape of the individual particles. The first three have been shown to affect coke properties, but the effects vary. The effect of the fourth factor cannot be clearly shown with the available data, however, certain apparent trends are pointed out for further consideration.

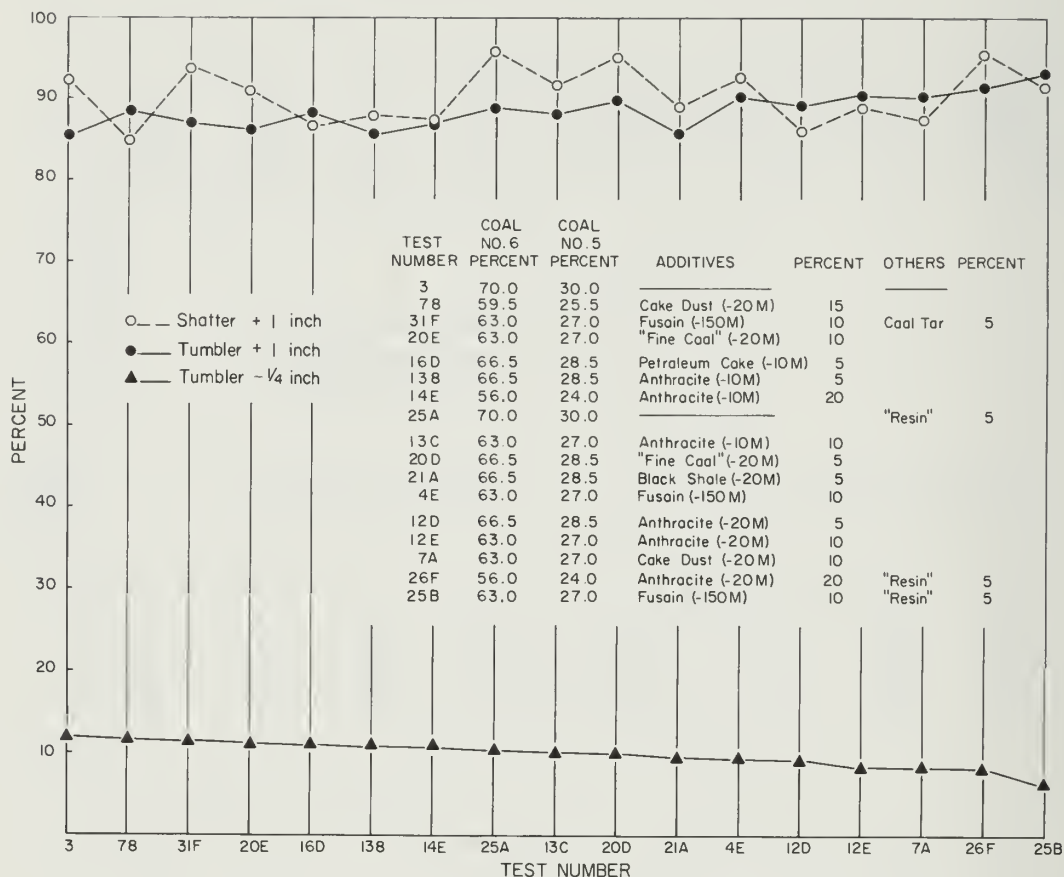


Fig. 16. Character of coke produced by adding various sizes and amounts of different additives to the standard sample.

Amount of the Additive

If any improvement occurred, coke quality generally improved with an additive, then declined as more additive was used. For example, coke produced by adding minus 150-mesh fusain to the coal charge showed the general improved trend, with a decline in coke quality as fusain was added after an optimum amount of 10 percent (fig. 7). Similar results are shown in blends with minus 150-mesh anthracite (fig. 8) and minus 20-mesh coke dust (fig. 9).

An exception is shown in figure 8, in which an optimum coke was produced with 10 percent minus 20-mesh anthracite added, a decline in indices occurred when 15 percent was added, then an improvement occurred as 20 percent anthracite was used. Another exception is shown in figure 11. The optimum tumbler indices of the coke was produced from 5 percent minus 10-mesh petroleum coke and the standard sample. The addition of 10 and 15 percent of the petroleum coke to the coal charge resulted in a decline of tumbler indices. As 20 percent minus 10-mesh petroleum coke was used in the mixture an upward trend in the tumbler indices was produced.

Particle Size of the Additive

Effect of particle size of the additives upon the coke may be seen by a study of figure 15. Each of the three charts in this figure represents coke data produced by blending various amounts of the additives of different size range with the standard sample. Parentheses around the percentage figures at the bottom of the chart identify those blends which produced cokes having better physical properties than the coke produced from the standard sample alone. These cokes are referred to as "improved cokes." Fusain in the minus 150-mesh size range, four blends of minus 10-mesh material, three anthracites (5, 10, and 20 percent), and one petroleum coke (5 percent) produced improved coke. Seven blends of minus 20-mesh material, coke dust (10 and 15 percent), anthracite (5 and 10 percent), fine coal (5 and 10 percent), and black shale (5 percent) also produced improved cokes.

Nature of the Additive

Although the nature of an additive is an important factor in coking, the characteristics that cause it to react differently from other additives are not clear. Chemical composition of the standard sample and the additive and the relation between them are probably important, and for these coking tests a rather large spread occurred between the different components used, especially in fixed carbon and volatile content.

A totally inert material does not shrink but it expands slightly upon heating, therefore the shrinkage of a mixture is reduced and fracturing is reduced. If the maximum expansion occurs after resolidification of the main mass, fractures will form which will weaken the coke. The possible different expansion characteristics of high-carbon components used in this investigation were not studied but should be considered.

The ability of the surface of the additive to adsorb or be wetted by the fluid derived from heating of the coal will affect the coking properties of the blend. The presence of pyrite is recognized as harmful because it increases the amount of sulfur in the metallurgical coke, but the effect of other minerals upon the structure of the coke needs to be investigated.

Shape of Individual Particles of Additive

An attempt to evaluate the effect of the shape of individual particles upon the coke character did not produce a clear correlation, but two observations were made.

Both shatter and tumbler indices show a great difference between coke produced from coke dust and from coke breeze in this investigation (table 5 and figs. 8, 9). Chemically these two additives vary slightly with the highest, dry, ash-free, fixed carbon found in the coke dust - 98 percent as compared to 94.5 for the coke breeze (table 3). Microscopically the two materials appear to be the same except for the shape of the individual particles (table 4). In the coke dust 3.8 percent of the particles were angular in shape; in the coke breeze a high of 15.8 percent of the individual particles were angular in shape and this breeze produced by far the poorest coke of the test series.

Similarly, the chemical composition of fusain from the No. 6 Coal and anthracite used in this investigation are about the same; anthracite had a fixed-carbon content of 93.1 and fusain had a fixed carbon content of 89.3 on the moisture-

and ash-free basis (table 3). Petrographically fusain and anthracite are similar, but not to the same degree as the coke dust and coke breeze; fusain can be readily distinguished from the anthracite but there is no essential petrographic difference between the coke dust and coke breeze used in this investigation. The high percentage (79.6 percent) of rodlike particles in fusain is striking. This size composition, minus 150-mesh, produced the optimum coke, as compared to other additives of the same size composition. Minus 150-mesh anthracite had only 21.9 percent rodlike particles. Possibly the rodlike character of the fine fusain tends to bind the viscous material into a mass.

SUMMARY AND CONCLUSION

Those additives that gave an improved coke when blended with the standard sample are summarized in figure 16. Tumbler plus 1-inch and tumbler minus 1/4-inch were considered the most important indices and were used primarily in evaluating the character of the coke. Shatter indices were not disregarded, however, and were used as a deciding factor in evaluating the relative quality of two cokes that had essentially the same tumbler indices. For completeness, all indices have been plotted on all diagrams but only the two tumbler indices mentioned above and the shatter plus 1-inch were plotted on figure 16.

The test numbers have been arranged to show a progressive decrease in the minus 1/4-inch fine material in cokes produced. Although the relative quality of cokes shown in adjacent tests in figure 16 may not differ greatly, this arrangement of tests shows that a decided improvement in coke character was produced by blending additives of certain nature, size, and amount with the standard sample.

In conclusion, this investigation has shown that the character of coke produced on a laboratory scale from Illinois coals can be improved by altering the petrographic composition of the coal charge with the addition of high-carbon and other components. Character of the coke produced from these blends depends upon a number of factors including the original petrographic composition and the particle size of the coal.

It has been shown that the amount, particle size, and nature of the additive affect the composition of the coke, and an interrelation appears to exist between these different factors. Possibly the shape of the individual particles of the additive also affect the character of the coke produced.

From these data it can be concluded that blending additives to a coal charge to produce an optimum coke is not a haphazard process. Many factors must be considered and each critically evaluated if an optimum coke is to be produced from any one mixture of coal and other components.

A logical continuation of this investigation would be on a pilot scale to determine if trends established in this laboratory scale study would be the same in the larger scale investigation. Certain problems exist such as securing a homogeneous mixture if great size variations prevail between the coal and the additive, hence minimizing segregation in handling the mixture and charging the coke oven is considered to be very important.

REFERENCES

- Cady, G. H., et al., 1952, Movable coal resources of Illinois: Illinois Geol. Survey Bull. 78.
- Chayes, F., 1949, A simple point counter for thin-section analysis, Am. Mineralogist, v. 34, no. 1 and 2, p. 1 - 11.
- Gregory, J. A. and Felton, D., 1959, Pilot plant research into methods of improving blast furnace coke quality; the effect of mineral matter in coal on the physical strength of blast furnace coke; pilot oven tests results with 1% of bentonite or bentonitic shale added to the coal charge: Notes on publication of the Work Research Department, The Broken Hill Proprietary Company Limited (Iron and Steel Works) Newcastle, Rept. no. M. R. 555.
- Harrison, John A., 1958, Modifications in petrographic composition of coal charges and their influence on the resulting coke (abstract): Geol. Soc. America Bull. 69, no. 12, pt. 2., p. 1578.
- Jackman, H. W., Eissler, R. L., and Reed, F. H., 1955, Comparison of mine sizes of southern Illinois coals for use in metallurgical coke: Illinois Geol. Survey Circ. 205.
- Jackman, H. W., Helfinstine, R. J., Eissler, R. L., and Reed, F. H., 1955, Coke oven to measure expansion pressure - modified Illinois oven: Am. Inst. Min. Met. Eng., Blast Furnace, Coke Oven and Raw Materials Conference, Philadelphia, Pa.; Survey reprint series 1955-E.
- Jackman, H. W., Henline, P. W., and Reed, F. H., 1956, Factors affecting coke size: Illinois Geol. Survey Circ. 213.
- Jackman, H. W., Eissler, R. L., and Reed, F. H., 1956, Coking coals of Illinois: Illinois Geol. Survey Circ. 219.
- Jackman, H. W., Eissler, R. L., and Helfinstine, R. J., 1958, Influence of coking time on expansion pressure and coke quality: Illinois Geol. Survey Circ. 246.
- Marshall, Charles E., Harrison, John A., Simon, Jack A., and Parker, Margaret A., 1958, Petrography and coking character of coal: Illinois Geol. Survey Bull. 84.
- Reed, F. H., Jackman, H. W., Rees, O. W., Yohe, G. R., and Henline, P. W., 1952, Some observations on the blending of coals for metallurgical coke: Illinois Geol. Survey Circ. 178.
- Rees, O. W., and Pierron, E. D., 1954, Plastic and swelling properties of Illinois coals: Illinois Geol. Survey Circ. 190.
- Rees, O. W., and Pierron, E. D., 1955, The effect of diluents on the plastic property of coal as measured by the Gieseler Plastometer: Illinois Geol. Survey Circ. 197.
- Thiessen, G., 1936, Fusain content of coal dust from Illinois dedusting plant: Am. Inst. Min. Met. Eng. Tech. Pub. 664, Class F Coal Div. No. 70.
- Reed, F. H., Jackman, H. W., Rees, O. W., Yohe, G. R., and Henline, P. W., 1947, Use of Illinois coal for production of metallurgical coke: Illinois Geol. Survey Bull. 71.

Illinois State Geological Survey Circular 289
29 p., 16 figs., 5 tables, 1960



CIRCULAR 289

ILLINOIS STATE GEOLOGICAL SURVEY

URBANA

